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**Dreher et al.**

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(54) **POMEGRANATE SEED OIL COMPOSITION**

USPC ..... 424/725, 776  
See application file for complete search history.

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(56) **References Cited**

U.S. PATENT DOCUMENTS

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4,728,522 A \* 3/1988 Wear et al. .... 426/242  
7,943,185 B1 \* 5/2011 Anderson et al. .... 424/776  
8,263,140 B1 \* 9/2012 Dreher et al. .... 424/725

(\*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 553 days.

FOREIGN PATENT DOCUMENTS

This patent is subject to a terminal disclaimer.

JP 2002121581 A \* 4/2002

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OTHER PUBLICATIONS

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Farr ((2000) Chapter 8: Refining Fats and Oils. In O'Brien (ed.) Introduction to Fats and Oils Technology (2nd edition, pp. 136-151) USA: AOCS Press).\*

\* cited by examiner

**Related U.S. Application Data**

(63) Continuation-in-part of application No. 13/082,343, filed on Apr. 7, 2011, now Pat. No. 8,889,199, which is a continuation-in-part of application No. 11/687,480, filed on Mar. 16, 2007, now Pat. No. 7,943,185.

*Primary Examiner* — Susan Hoffman

(60) Provisional application No. 60/888,763, filed on Feb. 7, 2007, provisional application No. 60/888,762, filed on Feb. 7, 2007.

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(51) **Int. Cl.**  
**A61K 36/00** (2006.01)

(57) **ABSTRACT**

(52) **U.S. Cl.**  
CPC ..... **A61K 36/00** (2013.01)

A stabilized seed oil composition that includes oil extracted from pomegranate seeds. The extracted oil has at least about 65% punicic acid. The extracted oil has reduced levels of free fatty acids compared to cold pressed pomegranate seed oil, reduced peroxide values compared to said cold pressed pomegranate seed oil, and a lower yellow color value compared to said cold pressed pomegranate seed oil.

(58) **Field of Classification Search**  
CPC ..... A61K 36/185; A61K 8/922

**16 Claims, 8 Drawing Sheets**

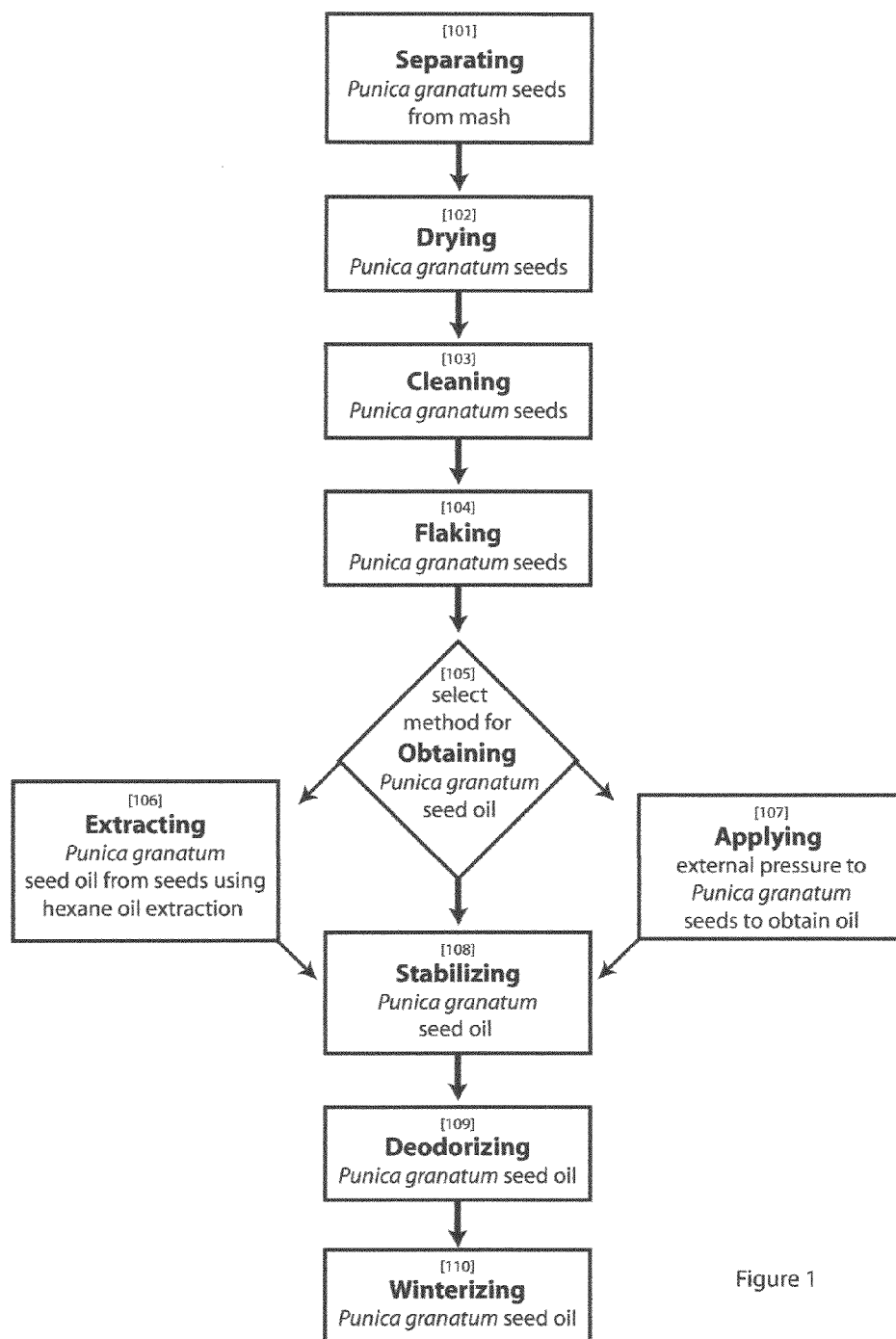


Figure 1

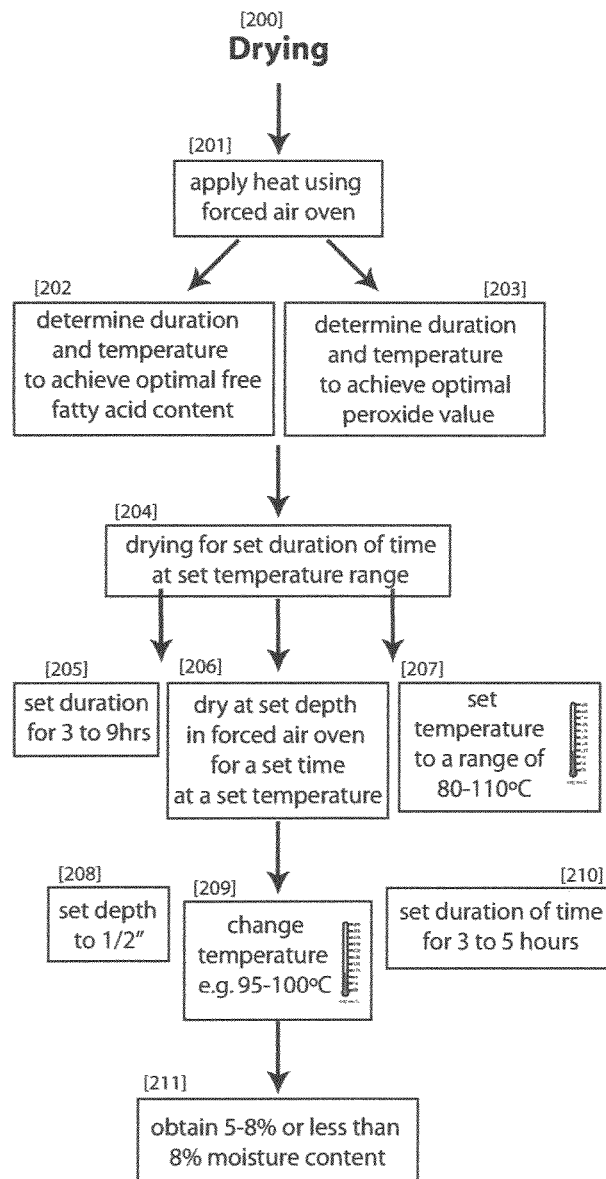


Figure 2

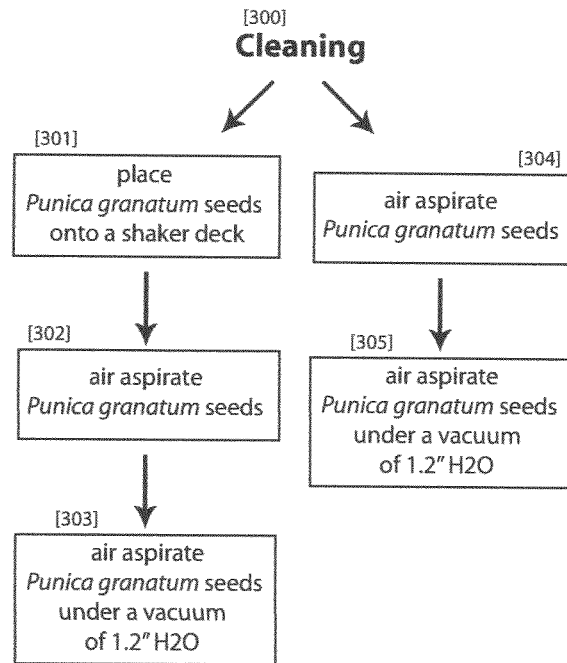


Figure 3

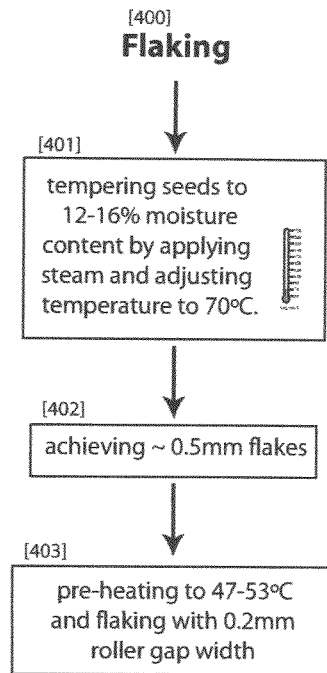


Figure 4

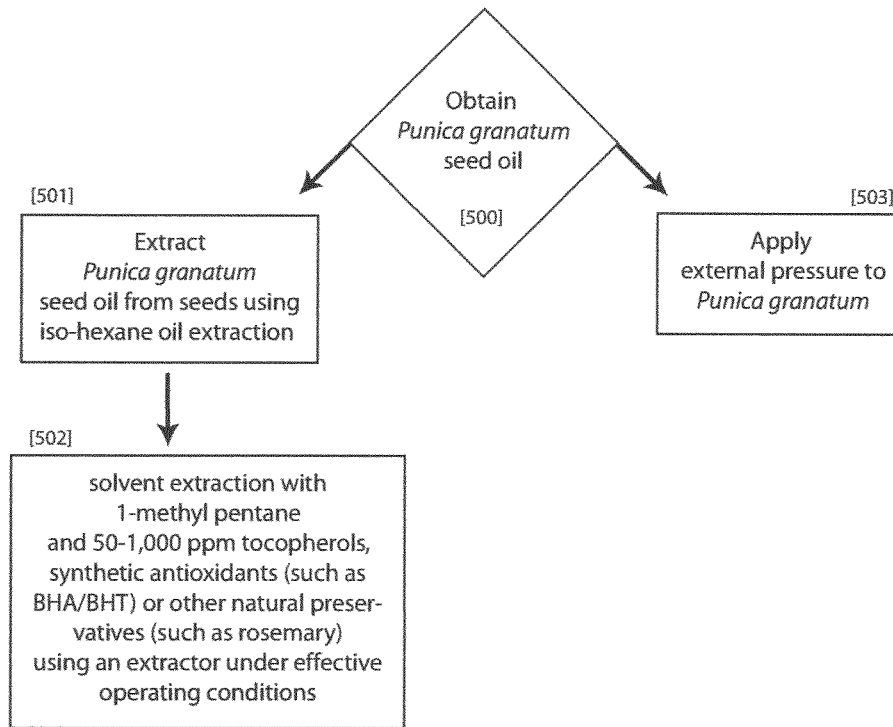


Figure 5

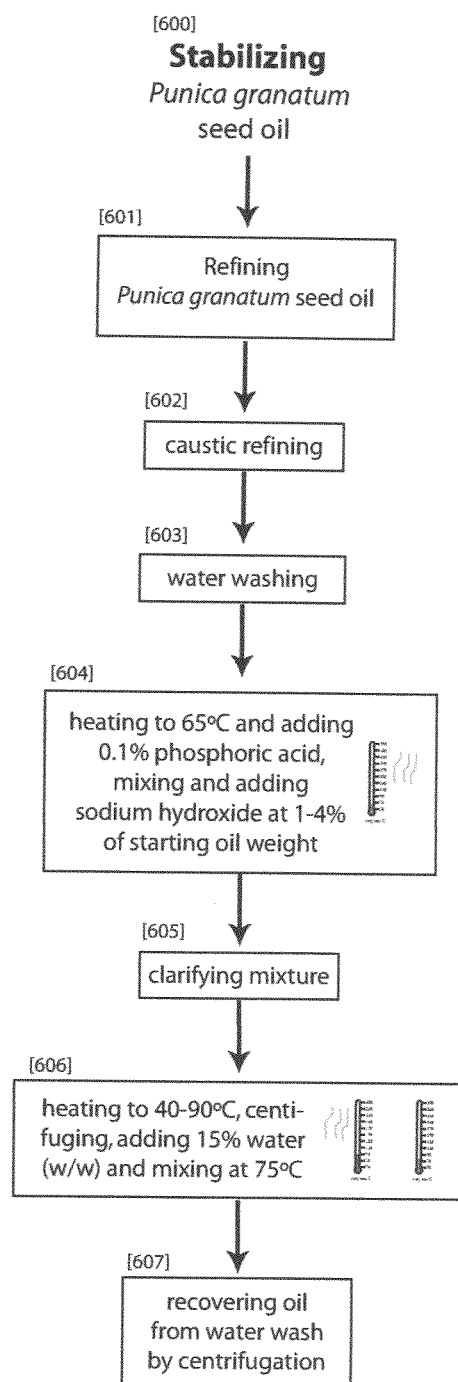


Figure 6

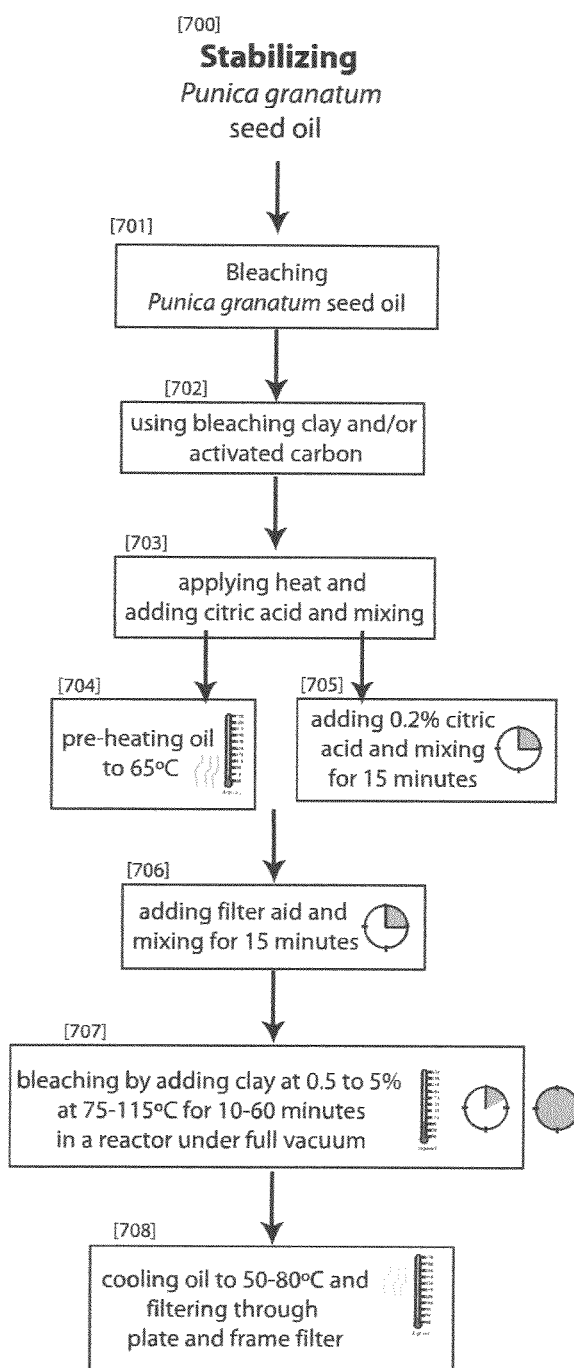


Figure 7



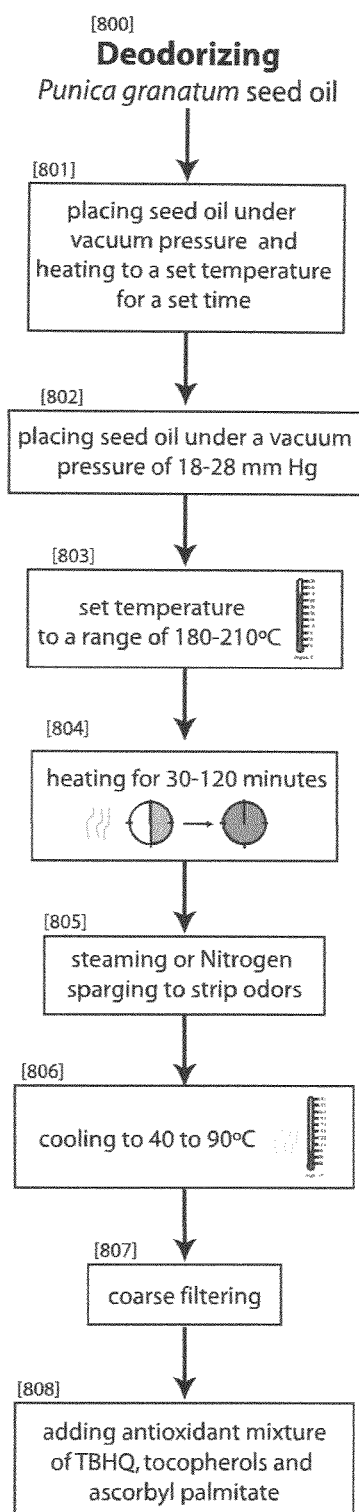


Figure 8

**POMEGRANATE SEED OIL COMPOSITION**

This application is a continuation-in-part of U.S. Utility patent application Ser. No. 13/082,343, filed Apr. 7, 2011, now U.S. Pat. No. 8,889,199, which is a continuation-in-part of U.S. Utility patent application Ser. No. 11/687,480 filed Mar. 16, 2007, now U.S. Pat. No. 7,943,185, which claims benefit of U.S. Provisional Patent Application Ser. No. 60/888,763 filed Feb. 7, 2007, and Ser. No. 60/888,762 filed Feb. 7, 2007, the contents of which are incorporated herein by reference in their entirety for completeness of disclosure.

**BACKGROUND OF THE INVENTION****1. Field of the Invention**

One or more embodiments of the invention relate to novel oil fractions extracted from *Punica granatum* (pomegranate) fruit, to the methods and processes for preparation thereof, to compositions containing them and the product created by the method. The invention relates generally to a process for isolating stabilized and deodorized pomegranate seed oil.

**2. Description of the Related Art**

There are existing processes for isolating oil from fruit seeds. However, pomegranate seed oil is unique. Cold press techniques for isolating pomegranate seed oil yield an unstable and vulnerable product. The product is readily susceptible to decay and goes rancid without hesitation. When applied to pomegranate seeds the standard deodorization and extraction processes for seed oil yields an unstable and readily polymerized product. The pomegranate seed product extracted using standard methods not only has an unpleasant odor but also can form into a gel-like substance making it difficult to use for a variety of products. Pomegranate seed oil has been described as having an "odd flavor" and a "putrid" or "unpleasant" smell. It is thought the high levels of the beneficial constituents in the pomegranate seed oil make it more reactive than other seed oils. Sometimes polymerized oil can be treated with hexane to turn it back into the desirable liquid state. However, this standard technique has proved ineffective with pomegranate seed oil.

Furthermore, since pomegranate seed oil is unstable and goes rancid fairly quickly it minimizes and limits the commercial practicability for the oil. The unstable and negative odor properties of the pomegranate oil results in a product that has little practical use in commercial products such as cosmetics, skin creams, eye creams, dietary supplements and other medicinal applications. Current processes do not yield a useful pomegranate oil product. The present invention defines a process wherein the negative properties of pomegranate seed oil extraction are ameliorated or sufficiently reduced while the beneficial constituents of pomegranate seed oil remain intact.

Pomegranate seed oil contains approximately 65% punicic acid, a conjugated linolenic acid (9 cis, 11 trans, 13 cis-CLNA) and has been reported to possess medicinal properties including anti-carcinogenic activity against breast cancer and skin cancer. The high polyunsaturated fatty acid content, however, makes this oil extremely susceptible to oxidative rancidity. As a result, extraction and refining process parameters typically employed for edible and cosmetic oils may degrade the pomegranate seed oil.

The benefits of pomegranate seed oil have been reported to act in the areas of immune function, cancer prevention and therapy, cardiovascular health, skin health, and healthy lipid metabolism.

Pomegranate seed oil has been mentioned as a treatment for various ailments. It has been shown that pomegranate seed

oil has benefits on immune function. Serum, liver, epididymal, and perirenal adipose punicic acid levels were high with increases in dietary pomegranate seed oil levels. However, punicic acid was not detected in splenocytes for any dietary group. The results suggest that pomegranate seed oil may enhance B-cell function in vivo.

Pomegranate seed oil has been implicated in the treatment and prevention of various cancers. Anti-carcinogenic and chemopreventive components have been found in pomegranate seed oil. Findings indicated the chemopreventive activity of pomegranate seed oil as well as an isolated HPLC fraction, referred to as peak B. An effect of pomegranate extracts on inhibiting prostate cancer cell proliferation has been described. The study shows the interactions of the extracts from three pomegranate compartments (peels, juice, seeds) in inhibiting prostate cancer cell proliferation, invasion and phospholipase A-2 (associated with invasive potential) expression. This study indicates the possibilities of pomegranate as a synergistic drug for the treatment of cancer.

Further studies show the potential and chemopreventive nature of pomegranate seed oil. Another study discusses the beneficial role of CLA-rich pomegranate seed oil in colon carcinogenesis. Topical application of pomegranate seed oils has been shown to be an effective chemopreventive treatment for skin cancer. Another study discusses the potential of pomegranate seed oil and polyphenols in fermented pomegranate juice to have an inhibitory effect on oxidation and prostaglandin synthesis, as well as inhibit breast cancer cell proliferation and invasion, and promote apoptosis of breast cancer cells. Further studies indicated pomegranate seed oils containing conjugated linolenic acids (CLN) were cytotoxic to human monocytic leukemia cells.

Pomegranate seed oil is also mentioned as a possible treatment for skin repair. Cosmeceutical applications for pomegranate suggest a potential for certain pomegranate fractions for facilitating skin repair, namely aqueous extracts (especially that from pomegranate peel) for promoting the repair of dermis, and pomegranate seed oil promoting regeneration of epidermis.

Pomegranate seed oil has been indicated in the maintenance of healthy lipid metabolism and cholesterol levels. The hypocholesterolemic activity of LN (linolenic acid) and CLN (conjugated linolenic acid) have been described. CLN is found in pomegranate seed oil and it had no effect on serum cholesterol. CLN and LN significantly decrease hepatic cholesterol but no effect was observed on heart and kidney cholesterol levels. A report indicates that pomegranate seed oil has an effect on lipid metabolism.

Pomegranate seed oil has been shown to have benefits for cardiac problems and even asthma. A potential role for pomegranate extracts as cardioprotective and an anti-inflammatory medicament for internal and/or external applications have been proposed for treatments such as asthma. Fermented pomegranate juice and cold pressed pomegranate fruit extract can reduce prostaglandin and leukotriene formation by the inhibition of cyclooxygenase and lipoxygenase activities. Leukotriene participates in allergic responses and particularly the bronchoconstriction involved in asthma. Pomegranate fractions have an anti-angiogenic potential. More cardio info.

The active components of the pomegranate fruit have begun to be isolated and identified. The presence of estrogenic compounds in pomegranate peel extracts was reported. In this study estrogenic compounds luteolin, quercetin, and kaempferol were detected and identified in the pomegranate peel.

One or more embodiments of the invention maintain the positive qualities of the pomegranate seed oil while minimizing and deleting the less desirable qualities such as unpleasant odor and a short shelf-life. Hence embodiments of the invention may be referred to generally as a process of stabilizing and deodorizing pomegranate seed oil.

#### BRIEF SUMMARY OF THE INVENTION

One or more embodiments of the pomegranate seed oil composition are directed to a stabilized seed oil composition including oil extracted from pomegranate seeds. The extracted oil has reduced levels of free fatty acids compared to cold pressed pomegranate seed oil. The extracted oil has reduced peroxide values compared to the cold pressed pomegranate seed oil. The extracted oil has lower yellow color value compared to the cold pressed pomegranate seed oil.

In one or more embodiments, the extracted oil includes at least about 65% puniceic acid.

The reduced levels of free fatty acids may be lower than about 1.54%. The reduced levels of free fatty acids may be less than about 0.5%, less than about 0.2%, or preferably less than about 0.1%. The reduced levels of free fatty acids may be between about 0.07% to about 0.03%.

The reduced peroxide values may be less than about 0.69 meq/kg. The reduced peroxide values is less than about 0.3 meq/kg, or preferably less than about 0.1 meq/kg.

The lower yellow color value may be less than a Lovibond Y value of less than about 70. The lower yellow color value may be less than a Lovibond Y value of less than about 15.0, preferably less than a Lovibond Y value of less than about 5.0, or less than a Lovibond Y value of less than about 2.0.

In one or more embodiments, the extracted oil includes a lower red color value compared to the cold pressed pomegranate seed oil. The lower red color value may be less than a Lovibond R value of less than about 2.9, or less than about 0.2.

The stabilized seed oil composition may also, further include between about 50 ppm to about 1,000 ppm added antioxidants. The added antioxidants may include tocopherols. The added antioxidants may include at least one of butylated hydroxyanisole (BHA), butylated hydroxytoluene (BHT), and tert-Butylhydroquinone (TBHQ), and ascorbyl palmitate. For example, the stabilized seed oil composition may include at least about 650 ppm tert-Butylhydroquinone (TBHQ), at least about 120 ppm tocopherols, and at least about 100 ppm ascorbyl palmitate.

The oil extracted from the pomegranate seeds may be stabilized. Stabilizing may include refining and bleaching. Stabilizing may further include winterizing. In one or more embodiments, the oil extracted from the pomegranate seeds is deodorized.

The oil extracted from the pomegranate seeds may be extracted from pomegranate seeds dried at a temperature of between about 80° C. to about 110° C. The oil extracted from the pomegranate seeds may be extracted from pomegranate seeds dried for a duration of between about 3 hours to about 9 hours. The drying temperature and/or duration may achieve one or more effects, including but not limited to moisture removal and affecting at least one of free fatty acid content and peroxide value.

One or more embodiments of the pomegranate seed oil composition are directed to a stabilized seed oil composition with oil extracted from pomegranate seeds. The extracted oil has a free fatty acid level of less than about 1.54%, a peroxide value of less than about 2.9 meq/kg, and a Lovibond Y value of less than about 70.

One or more embodiments of the pomegranate seed oil composition are directed to a stabilized seed oil composition with oil extracted from pomegranate seeds. The extracted oil has a free fatty acid level of less than about 0.01%, a peroxide value of less than about 0.1 meq/kg, and a Lovibond Y value of less than about 5.

One or more embodiments of the invention are directed to a method, a composition and a product by process for manufacturing a stabilized form of pomegranate seed oil.

One or more embodiments of the pomegranate seed oil composition are directed to a stabilized seed oil composition including oil extracted from pomegranate seeds. The extracted oil includes at least about 65% puniceic acid. The extracted oil has reduced levels of free fatty acids compared to cold pressed pomegranate seed oil. The extracted oil has reduced peroxide values compared to the cold pressed pomegranate seed oil. The extracted oil has lower yellow color value compared to the cold pressed pomegranate seed oil.

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One or more embodiments of the invention are directed to a method for extracting oil from *Punica granatum* seeds to formulate a stable and deodorized pomegranate seed oil. The method generally directed to a process that involves separating, drying, cleaning, flaking, and selecting a method for obtaining the *Punica granatum* seeds.

The process of separating is the initial step and generally makes use of any acceptable method for separating the *Punica granatum* seeds from a mash.

The drying step is accomplished by applying heat to the seeds through the use of a forced air dryer or other mechanism for reducing the water level within the seeds. When using the forced air embodiment drying is initiated for a duration and temperature sufficient to achieve optimal peroxide value and/or optimal free fatty acid content. This is generally accomplished by drying the seeds for a set duration of time at a set temperature range. For instance, the drying can occur for a set duration of 3 to 9 hours and the set temperature range may be between 80-110° C. Drying of the *Punica granatum* seeds may occur by placing *Punica granatum* seeds at a set depth in a forced air dryer for the set time at a set temperature. The set depth of forced air oven may be ½". The set temperature may be between 95-100° C. And the set time may range between 3 to 5 hours. Generally drying is performed until the *Punica granatum* seeds contain 5-8% moisture content or less than 8% moisture content.

The cleaning step generally involves cleaning the *Punica granatum* seeds via a shaker deck and air aspiration. When air aspiration is used it is typically done under a vacuum of 1.2" of water. Other methods of cleaning the seeds are however still within the scope and spirit of the invention and any such means that accomplishes this goal is acceptable.

Flaking of the *Punica granatum* seeds comprises tempering the seeds from 16% moisture content by applying steam and adjusting the temperature to 70° C. Flaking the *Punica granatum* seeds may produce flakes of approximately 0.5 mm in thickness and is accomplished in at least one embodiment of the invention by pre-heating seeds to 50±3° C. in a cooker section of a cooker pre-press then flaking using a roller gap width setting of 0.2 mm.

Selection of a method for obtaining *Punica granatum* seed oil may comprise extracting of seed oil from the *Punica granatum* seeds using solvent extraction using 1-methyl pentane. on a counter-current extractor. The extraction may be immediately mixed with 50 to 1,000 ppm mixed tocopherols. Other natural antioxidants (e.g. Rosemary) or synthetic antioxidants (e.g. BHA/BHT) may also be used. The selecting of method for obtaining *Punica granatum* seed oil may comprise extracting of seed oil by applying external pressure to the *Punica granatum* seeds.

Stabilizing of the *Punica granatum* seed oil generally comprises a refining and bleaching step. Refining may be achieved by caustic refining and/or refining by water washing. Stabilizing of the *Punica granatum* seed oil may comprise refining by heating to 65° C. and adding 0.1% phosphoric acid, mixing and adding sodium hydroxide at 1 to 4% of starting oil weight. Stabilizing of the *Punica granatum* seed oil may comprise refining by clarifying mixture by heating to 40 to 90° C. and centrifuging adding 15% water (w/w) and mixing at 75° C. The stabilizing of the *Punica granatum* seed oil may comprise refining by recovering oil from water wash by centrifugation and involve a bleaching step that makes use of bleaching clay or any other acceptable bleaching means. Bleaching may occur by applying heat, adding citric acid and mixing. Stabilizing of the *Punica granatum* seed oil may make use of bleaching by pre-heating oil to 65° C. The stabilization step may also involve bleaching by adding 0.2% citric acid and mixing for 15 minutes. Achieving stabilization may generally involve bleaching by using a cellulose-acetate filter aid and adding clay or activated carbon and mixing for 15 minutes. The bleaching step may involve adding clay at 0.5% to 5% or activated carbon at 75-115° C. for 10 to 60

minutes in a reactor under full vacuum; and bleaching by cooling oil to 65° C. and filtering through filter elements.

Deodorizing the *Punica granatum* seed oil is also part of the process and may comprise putting the *Punica granatum* seed oil under vacuum pressure and heating to a set temperature for a set duration of time. Deodorizing of the *Punica granatum* seed oil involves placing the bleached seed oil under of vacuum pressure of 18-28 mm Hg, heating the seed oil to a temperature range between 180-210° C. for a range of approximately 30 to 120 minutes and steaming and/or nitrogen sparging. A cooling step is then performed that involves cooling the seed oil to 40-90° C. and filtering through a coarse filter. An antioxidant mixture of TBHQ, tocopherols, and ascorbyl palmitate may also be added to the seed oil as part of the process described herein.

#### BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 illustrates the method of isolating and obtaining, stabilizing and deodorizing *Punica granatum* seed oil.

FIG. 2 illustrates the method of drying the seeds of *Punica granatum*.

FIG. 3 illustrates the method of cleaning the seeds of *Punica granatum*.

FIG. 4 illustrates the method of flaking the seeds of *Punica granatum*.

FIG. 5 illustrates the method of obtaining the seed oil from the seeds of *Punica granatum*.

FIG. 6 illustrates the method of stabilizing *Punica granatum* seed oil.

FIG. 7 further illustrates the method of stabilizing *Punica granatum* seed oil.

FIG. 8 illustrates the method of deodorizing *Punica granatum* seed oil.

#### DETAILED DESCRIPTION OF THE INVENTION

The following descriptions of one or more embodiments of the invention are exemplary, rather than limiting, and many variations and modifications are within the scope and spirit of the invention. Although numerous specific details are set forth in order to provide a thorough understanding of the present invention, it will be apparent to one of ordinary skill in the art, that embodiments of the invention may be practiced without these specific details. In other instances, well-known features have not been described in detail in order to avoid unnecessarily obscuring the invention which is directed to processes for the preparation of pomegranate seeds and the extraction, stabilization, and deodorization of pomegranate seed oil. The processes provide a technique wherein the beneficial components of pomegranate seed oil remain intact subsequent to processing.

One or more embodiments of the invention are directed to a unique method, composition and a product by process for pomegranate seed oil.

Generally, as depicted in an overview in FIG. 1, the processes of the invention are characterized by the following steps:

Separating [101], Drying [102], Cleaning [103], Flaking [104], Obtaining [105] by extracting [106] or applying pressure [107], Stabilizing [108], and Deodorizing [109].

These steps are depicted in further detail throughout the application.

As used herein "polyphenols" refers to or means but not is limited to a polyhydroxy phenol; especially an antioxidant phytochemical that tends to prevent or neutralize the damaging effects of free radicals.

As used herein, the terms “reduces,” “reduced,” or “reducing,” when used to refer to a particular odor or taste characteristic, refers to any observable lessening of that characteristic when the method or composition of the present invention is compared to prior art methods or compositions.

As used herein, the terms “odor,” “smell,” and “scent” refer to olfactory recognition of any particular or group of particular gases. Preferably, the olfactory recognition refers to mammalian, and most preferably human, olfactory recognition. Such recognition was determined by observation by the inventors, and may alternatively be determined by a cross-section of the population or by quantitative or qualitative recognition devices known to those of skill in the art.

SEPARATING. Separating is shown in the first block of FIG. 1 [101]. One or more embodiments of the invention involve separating pomegranate seeds from a mash of pomegranate fruit [101].

DRYING. The drying step is shown in block 2 of FIG. 1 [102] and further depicted in detail in FIG. 2 [200]. One or more embodiments of the invention involve a drying step for purposes of removing moisture from the pomegranate fruit seeds from which the seed oil is to be extracted. Moisture removal may be carried out using any acceptable process but is achieved in at least one embodiment of the invention by placing pomegranate seeds in a forced air dryer [201]. Moisture removal may occur for a duration and temperature to achieve optimal free fatty acid content [202] and/or optimal peroxide value [203]. Moisture removal may occur for a set duration of time at a set temperature range [204] and occur at a set depth in a forced air dryer for a set time at a set temperature [206]. The set temperature range of 80-110° C. [207] is generally acceptable for achieving the proper level of drying when drying is done for a duration of 3 to 9 hours [205]. More specifically but not by way of limitation moisture removal may be carried out by placing the pomegranate seeds in a forced air tunnel dryer at a depth of ½" at 100±5° C. for a time of 3 to 5 hours to yield seeds with 5-8% or less than 8% moisture content [208, 209, 210, 211].

CLEANING. The cleaning step is shown in block 3 of FIG. 1 [103] and further depicted in detail in FIG. 3 [300]. Dried pomegranate seeds can contain dried pulp and skin fragments. It is generally beneficial to remove these unwanted materials from the pomegranate seeds via a cleaning step. Removal of unwanted dried material is carried out in at least one embodiment of the invention by placing *Punica granatum* seeds onto a shaker deck [301]. An air aspirator [302, 304] is also helpful when seeking to remove these unwanted materials. When using an air aspirator application is typically performed under a vacuum of 1.2" of water [303] to yield cleaned dried seed free of pulp and skin fragments. The removal of unwanted material after solvent oil extraction can yield oil that is lighter in color requiring a less rigorous bleaching regimen. To summarize the removal of unwanted dried material from the seeds may be carried out by placing seeds onto a shaker deck, air aspiration, and air aspiration under a vacuum or 1.2" of water [301, 302, 303].

FLAKING. The flaking step is shown in block 4 of FIG. 1 [104] and further depicted in detail in FIG. 4 [400]. The conversion of pomegranate seed into particles may be referred to as flaking [104, 400]. The conversion into particles may occur prior to solvent extraction with pomegranate seeds tempered to 12-16% moisture content [401] by using steam and adjusting temperature to 70° C. The flaking may produce ~0.5 mm flakes [402]. Flaking is carried out in accordance with one or more embodiments of the invention by heating the pomegranate seed to a temperature between 47-53° C. [403]. Pre-heating the pomegranate seed to 50±3° C. in the cooker

section of a cooker pre-press is generally advisable followed by flaking immediately thereafter using a roller gap width setting of 0.2 mm or other setting acceptable to accomplish the goal of flaking [403]. In the case that seed flaking results in excessive production of fines the moisture content may be adjusted by applying steam and changing the temperature to 70° C. For instance, ideal flaking conditions may produce large consistent flakes, 0.50±0.5 mm in thickness and 13.0±0.5% moisture content. From a 325 kg batch of tempered seed a yield of 310.9 kg of flaked pomegranate seed may be produced.

OBTAINING. The obtaining step is shown in block 5 of FIG. 1 [105] and in further detail in FIG. 5 [500]. Drawing forth of seed oil from flaked pomegranate seed is generally accomplished by applying external pressure [107, 503] and/or through the use of solvent extraction using iso-hexane [106, 501]. For example, 1-methyl pentane (isohexane) [502] generally serves as a reliable solvent for extracting the seed oil. The drawing forth of seed oil from flaked pomegranate seed may be carried out by solvent extraction using 1-methyl pentane (isohexane). The extraction may be immediately mixed with 50-1,000 ppm tocopherols or other natural antioxidants such as Rosemary or synthetic antioxidants such as BHA/BHT [502]. The solvent extraction may be carried out using an extractor (such as a Soxhlet) wherein pomegranate seed flakes can be loaded in to the screen sample holder and extracted for four hours to result in an extraction yield of greater than 95%. The solvent extraction may be carried out in 1-methyl pentane on a counter-current extractor (such as Crown) under effective operating conditions. The effective operating conditions for a counter-current extractor may be the following: Flake feed rate 65 Kg/h, Retention time of 60 minutes, Bed level setting at minimum, fresh solvent flow 150±5 Kg/h, Solvent temperature 52±4° C., RFE feed rate 21 pm, RFE steam pressure 13 psi, DT sparge steam off, DT top tray temperature 84±3° C., DT bottom tray temperature 100±2° C. The solvent extraction may contain the addition of mixed tocopherols to 1-methyl pentane to minimize oxidation of the oil. Mixing in tocopherols or other antioxidants may be added at a quantity of 50 to 1,000 ppm. The extraction may yield crude oil that has a PV of 0.69, FFA of 1.54%, and Lovibond color parameters (1 inch sample cell) 70.0 Y and 2.9 R.

STABILIZING (refining). The stabilizing step is shown in FIG. 1 [108] and in further detail in FIG. 6 and FIG. 7. Stabilizing has two major components refining, shown in FIG. 6 [601], and bleaching, shown in FIG. 7 [701]. One or more embodiments of the invention are directed to a unique process for stabilizing isolated *Punica granatum* seed oil [108, 600, 700]. FIG. 6 depicts further detail of the refining step of the stabilizing process [600]. The general objective in refining is to free the extracted pomegranate seed oil from impurities and/or unwanted material [601]. The impurities may be caustic, black sediment, or otherwise undesirable material. The process for freeing the impurities is generally referred to as refining or caustic refining [602]. Stabilizing the *Punica granatum* seed oil may require caustic refining. Crude oil extracted from pomegranate seed flakes may contain undesirable material. The stabilization may involve water washing [603] and require assessment of the need for refining the oil; Free fatty acid (FFA) values may indicate a need for caustic refinement of the oil. FFA values may be near 1.54% indicating a need for further refinement. Such refining is generally carried out by heating the crude pomegranate seed oil to 65° C. and adding 0.1% phosphoric acid. The mixture of crude pomegranate seed oil and phosphoric acid may then be mixed for 15 minutes and sodium hydroxide (18 Be) may be

added at 1-4% of the starting oil weight [604]. The mixture may then be clarified [605] by heating to 40 to 50° C. and applying centrifugation [606] to remove undesirable black sediment and other impurities from the oil. After the clarifying 15% (w/w) may be added and mixed for 15 minutes at 75° C. [606]. Oil may be recovered from water wash by centrifugation [607]. This process may yield an FFA value of the caustic refined and water washed oil of less than 0.5%. The refining process may include a winterization step wherein refrigeration is applied to facilitate wax removal.

STABILIZING (Bleaching). As previously stated stabilizing requires a two-fold process of refining and bleaching. FIG. 7 depicts further detail of the bleaching aspect of the stabilizing process [700] which generally lightens or removes color from the pomegranate seed oil [701]. This process of lightening or removal of color is generally referred to as bleaching [701]. The bleaching may be carried out using bleaching clay or activated carbon [702], applying heat and adding citric acid and mixing [703] and pre-heating oil to 65° C. [704]. Quantities of 0.2% citric acid are typically effective when mixing [705]. A filter aid (e.g. cellulose acetate) and mixing [706] is also beneficial. When adding clay 0.5-5% clay at 75-115° C. for 10-60 minutes in a reactor under full vacuum [707] is effective. Cooling the oil to 50 to 80° C. and filtering [708] also produces results. While any means for bleaching is acceptable, bleaching is generally carried out in a reactor (such as Parr) using various grades of bleaching clay and may undergo the following exemplary steps. Oil may be pre-heated to 65° C. and 0.2% citric acid may be added and mixed for 15 minutes. Trisyl S615 filter aid (0.2% w/w) may be added and mixing continued for another 15 minutes. Bleaching clay (3% w/w) may then be mixed into the oil and the reactor sealed. Bleaching may be performed at 95° C. for 30 minutes under full vacuum. The oil may then be cooled to 65° C. and filtered. The bleaching may be carried out using bleaching clay such as Tonsil Supreme 124FF and undergo the following steps: Oil can be heated to 65° and 0.2% citric acid added and mixed for 15 minutes. Trisyl 5615 filter aid (0.2%, w/w) can be added and mixing continued for an additional 15 minutes. Bleaching clay (3%, w/w) may then be mixed into the oil and the reactor sealed. Bleaching may be performed at 95° C. for 30 minutes under full vacuum in a mini-RBD bleaching reactor. The oil may then be cooled to 65° C. and filtered. The above mentioned steps may effectively lighten the color of the oil to achieve Lovibond Y and R values of 2.0 and 0.2, respectively.

DEODORIZATION. Deodorization is shown in FIG. 1 [109] and in FIG. 8 in further detail [800]. One or more embodiments of the invention make use of a deodorizing step to remove unpleasant qualities of pomegranate seed oil such as its offensive odor when extracted in raw form. The elimination or prevention of offensive odor of pomegranate seed oil is referred to as deodorizing. FIG. 8 depicts the process of deodorizing in detail [800]. Deodorizing is generally carried out by placing seed oil under vacuum pressure (e.g., 18-28 mm Hg [802]) and heating the seed oil to a set temperature (e.g., 180-210° C. [803]) for a set time [801] such as 30-120 minutes and sparging using steam or nitrogen gas [804, 805]. The deodorizing step also involves cooling the seed oil to 40-90° C. and coarse filtering [806, 807]. An antioxidant mixture such as tert-butylhydroxyquinone (TBHQ) at 50-200 ppm, tocopherols at 50-200 ppm and/or ascorbyl palmitate at 50-200 ppm [808] is generally helpful. Other quantities of tocopherol, TBHQ and ascorbyl palmitate are however also within the scope and spirit of the invention. The elimination or prevention of offensive odor of pomegranate seed oil may be carried out by heating the oil using a heating apparatus and

vacuum to a target temperature range below 210° C. The deodorization may occur by taking bleached pomegranate seed oil under vacuum pressure (28-30 mm Hg) and heating it to a temperature between 180-210° C. for 30 to 120 minutes. The preferred deodorization residence time range is 30 to 60 minutes.

The present invention provides a novel process that allows for the preparation of pomegranate seeds and the subsequent extraction, stabilization and deodorization of the polyphenol rich pomegranate seed oil.

The present method comprises a number of separate phases, which, taken together, produce the beneficial result. However, it will readily be appreciated that the division of the method into separate phases is an artificial construct, primarily to aid in the clarity of presenting the invention, and that various aspects of any particular phase may be performed out of the described order, or combined with aspects ascribed to other phases, while still obtaining the benefits of the invention.

The method for making pomegranate seed oil includes the processing steps of separating, drying, air aspiration, flaking, obtaining seed oil, stabilizing which includes refining and bleaching and deodorization.

## DEFINITIONS

As used herein "pomegranate seed oil" refers to or means a substance derived and prepared from the components of the pomegranate seed. The substance may be an extract of the pomegranate seed wherein it is a powder, a liquid, a juice, mixed in a solution, a pill, a solid, from a raw pomegranate fruit, from a fermented pomegranate fruit. The extract may contain enhanced polyphenols beyond that which is naturally within the extracted substances. The extract may contain enhanced conjugated linolenic acids, including but not limited to puniceic acid.

## Example 1

### Crude Oil Data Samples

The extraction process may affect the characteristics of crude pomegranate seed oil extracted from pomegranate seeds. Crude oil content may also differ based on individual crop characteristics, storage duration, and storage methods. In one instance, a crude extraction yielded crude oil with a peroxide value (PV) of 0.69, free fatty acid content (FFA) of 1.54%, and Lovibond color parameters (1 inch sample cell) 70.0 Y and 2.9 R.

TABLE 1

crude pomegranate seed oil analysis				
Crude Oil Data	Sample 1	Sample 2	Sample 3	Sample 4
Peroxide value (meq/kg)	.69	0.40	2.90	0.40
FFA (%)	1.54	0.59	0.38	0.37
Colour (1") - red	2.9	1.9	1.8	2.3
Colour (1") - yellow	70.0	70.0	70.0	70.0

## Example 2

### Effects of Refining and Bleaching on Select Samples

The effects of the refining and bleaching processes were evaluated based on Sample 4. Table 2 shows the composition of Sample 4 after at least one process described herein:

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TABLE 2

processed pomegranate seed oil analysis				
Processes:	Crude Oil Data	Refined/ TriSyl Treated	Bleached	Refined, Bleached, Deodorized
Peroxide value (meq/kg):	0.40	0.16	<0.10	<0.10
FFA (%):	0.37	0.11	0.09	0.18
Colour (1") - red:	2.3		3.8	0
Colour (1") - yellow:	70.0		70.0	3.4

## Example 3

## Pomegranate Seed Oil Compositions

Four exemplary pomegranate seed oil compositions comprising extracted oil from pomegranate seeds were analyzed. The analysis may also differ based on individual crop characteristics, storage duration, storage methods, variations in extraction, and other factors.

TABLE 3

crude pomegranate seed oil analysis (refined, bleached, deodorized)				
	Sample A	Sample B	Sample C	Sample D
FFA (%)	0.03	0.04	0.07	0.18
Colour (5¼") - red	1.5	2	1.3	0
Colour (5¼") - yellow	15	14	18	3.4
OSI rancimat @ 80° C. (hours)	12.2	13.2	15.4	13.4
GC-MS analysis, (ppm)				
Pentanal	1.8	1.3	<1.0	<1.0
Propanal	<1.0	<1.0	not measured	<1.0
Hexanal	<1.0	<1.0	<1.0	<1.0
Elemental analysis, (ppm)				
Silver	<0.05	missed	0.07	<0.05
Cadmium	<0.03	<0.03	<0.03	<0.03
Copper	<0.05	<0.05	<0.05	<0.05
Mercury	<0.20	<0.20	<0.20	<0.20
Lead	<0.20	<0.20	<0.20	<0.20
Zinc	<0.02	<0.02	<0.02	<0.02
Fatty acid composition (area %)				
C16 Palmitic	2.56	2.54	2.76	2.90
C16:1 Hexadecenoic	0.03	0.03	0.04	0.05
C17 Margaric	0.05	0.05	0.06	0.05
C18 Stearic	1.85	1.87	2.02	2.03
C18:1n9 Oleic	4.22	4.21	4.64	4.45
C18:1 Octadecenoic	0.43	0.44	0.44	0.47
C18:2 Linolenic	4.91	5.04	5.37	5.78
C18:2n6 Linoleic	—	—	—	—
C18:3 (9,11,13-Linolenic)	72.58	74.69	70.59	70.79
C18:3 CLNA isomers	9.46	6.93	9.55	9.29
C18:3n3 alpha-Linolenic	0.04	—	0.05	0.05
C18:3n6 gamma-Linolenic	—	—	—	—
C18:4n3	—	—	—	0.07
Octadecatetraenoic	—	—	—	—
C20 Arachidic	0.43	0.50	0.53	0.47
C20:1 Eicosenoic	0.73	0.75	0.80	0.76
C20:2n6 Eicosadienoic	—	0.04	—	—
C22 Behenic	0.09	0.09	0.10	0.09
C24 Lignoceric	0.05	0.06	0.06	0.05
Others	0.57	0.56	2.99	0.42
C18:2 CLA Isomers	2.00	2.20	2.37	2.28

Thus, while embodiments and alternatives have been disclosed and discussed, the invention herein is not limited to the particular disclosed embodiments or alternatives but encompasses the full breadth and scope of the invention including

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equivalents, and the invention is not limited except as set forth in and encompassed by the full breadth and scope of the claims herein:

What is claimed is:

1. A stabilized pomegranate seed oil composition comprising:

oil extracted from pomegranate seeds by a process comprising:

separating pomegranate seeds;  
drying said pomegranate seeds;  
cleaning said pomegranate seeds;  
tempering said seeds to 12 to 16% moisture content by applying steam and adjusting temperature to about 70° C.;

flaking said pomegranate seeds;

extracting said pomegranate seed oil from said flaked pomegranate seeds using solvent extraction process on a counter-current extractor using iso-hexane and mixed with antioxidants selected from at least one from a group consisting of tocopherols, natural antioxidants, and synthetic antioxidants; and  
stabilizing said pomegranate seed oil using water washing and caustic refining; and

wherein said oil composition has reduced levels of free fatty acids compared to cold pressed pomegranate seed oil;

wherein said oil composition has reduced peroxide values compared to said cold pressed pomegranate seed oil, and

wherein said oil composition has a lower yellow color value compared to said cold pressed pomegranate seed oil.

2. The stabilized seed oil composition of claim 1, wherein said oil composition comprises at least about 65% punicic acid.

3. The stabilized seed oil composition of claim 1, wherein said reduced levels of free fatty acids is lower than about 1.54%.

4. The stabilized seed oil composition of claim 1, wherein said reduced levels of free fatty acids is less than about 0.5%.

5. The stabilized seed oil composition of claim 1, wherein said reduced levels of free fatty acids is less than about 0.1%.

6. The stabilized seed oil composition of claim 1, wherein said reduced levels of free fatty acids is between about 0.07% to about 0.03%.

7. The stabilized seed oil composition of claim 1, wherein said reduced peroxide values is less than about 0.69 meq/kg.

8. The stabilized seed oil composition of claim 1, wherein said reduced peroxide values is less than about 0.1 meq/kg.

9. The stabilized seed oil composition of claim 1, wherein said lower yellow color value is less than a Lovibond Y value of less than about 70.

10. The stabilized seed oil composition of claim 1, wherein said lower yellow color value is less than a Lovibond Y value of less than about 5.0.

11. The stabilized seed oil composition of claim 1, wherein said oil composition comprises a lower red color value compared to said cold pressed pomegranate seed oil.

12. The stabilized seed oil composition of claim 11, wherein said lower red color value is less than a Lovibond R value of less than about 2.9.

13. The stabilized seed oil composition of claim 11, wherein said lower red color value is less than a Lovibond R value of less than about 0.2.

14. The stabilized seed oil composition of claim 1, wherein said oil extracted from said pomegranate seeds is extracted

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from pomegranate seeds dried at a temperature of between about 80° C. to about 110° C. for a duration of between about 3 hours to about 9 hours.

15. A stabilized pomegranate seed oil composition comprising:

oil extracted from pomegranate seeds by a process comprising:

separating pomegranate seeds;

drying said pomegranate seeds;

cleaning said pomegranate seeds;

flaking said pomegranate seeds, wherein said flaking comprises tempering said seeds to 12 to 16% moisture content by applying steam and adjusting temperature to about 70° C. and passing said tempered seeds through a roller with a gap width of approximately 0.2 mm;

extracting said pomegranate seed oil from said flaked pomegranate seeds using solvent extraction process on a counter-current extractor using iso-hexane and mixed with antioxidants selected from at least one from a group consisting of tocopherols, natural antioxidants, and synthetic antioxidants;

stabilizing said pomegranate seed oil mixture using water washing and caustic refining; and

deodorizing said stabilized pomegranate seed oil; and bleaching said deodorized and stabilized pomegranate seed oil.

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16. A stabilized pomegranate seed oil composition comprising:

oil extracted from pomegranate seeds by a process comprising:

separating pomegranate seeds;

drying said pomegranate seeds;

cleaning said pomegranate seeds;

flaking said pomegranate seeds, wherein said flaking comprises tempering said seeds to 12 to 16% moisture content by applying steam and adjusting temperature to about 70° C. and passing said tempered seeds through a roller with a gap width of approximately 0.2 mm;

extracting said pomegranate seed oil from said flaked pomegranate seeds using solvent extraction process on a counter-current extractor using iso-hexane and mixed with antioxidants selected from at least one from a group consisting of tocopherols, butylated hydroxyanisole (BHA), butylated hydroxytoluene (BHT), and tert-Butylhydroquinone (TBHQ), and ascorbyl palmitate;

stabilizing said pomegranate seed oil mixture using water washing and caustic refining; and

deodorizing said stabilized pomegranate seed oil; and

bleaching said deodorized and stabilized pomegranate seed oil.

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